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R-7284

(Unclassified Title)

FINAL REPORT,
RESEARCH ON THE RESPONSE OF
REINFORCED SOLID PROPELLANTS

By

A. A. Caputo
B. H. Minnich
E. L. Alexander

November 1967

Contract Nonr 4988(00)FBM
Task No. NR 064-469

Sponsored By
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Group 4
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REINFORCED SOLID PROPELLANTS

Group 4
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Contract Nonr 4988(00)FBM
Task No. NR 064-469

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DATE 30 November 1967

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FOREWORD

This final report covering the period of 1 June 1966 through 30 September 1967 was prepared by the Materials Research Group of the Research Division of Rocketdyne, a Division of North American Rockwell Corporation, in accordance with the requirements of Contract No. Nonr 4988(00)FBM. It is being submitted to the Head, Structural Mechanics Branch, Office of Naval Research, Washington, D. C., Attention: Mr. J. Crowley.

The authors wish to thank the following individuals of the Rocketdyne Research Division for their special contributions to this investigation: F. P. Hollenbach--Specimen and test fixture preparation and assistance in testing, and J. R. Fulton--Photography. Mr. J. M. Crowley of ONR provided important guidance in this investigation.

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ABSTRACT

Research on the structural behavior of wire-reinforced propellants is reported. Values for certain orthotropic material constants which should be useful in grain design have been determined experimentally. The effects of winding angle and wire modulus on the material response were investigated.

A problem in delamination of thick-wall wire-reinforced grains was encountered during another Navy program. Various means were therefore investigated in the course of this program to eliminate delamination. Use of an ambient-cure matrix to eliminate thermal stress during the cure cycle and two approaches to adding radial wire reinforcing were explored briefly. The most promising approach appeared to be through use of the new ambient-cure matrix which would be specifically tailored to reinforced propellants.

The structural response of an aluminum wire-reinforced grain that was designed to demonstrate deep submergence rocket launch applications was obtained. This grain which embodied a new concept in burning rate control, a perforated inner core, withstood 1920-psi external pressure before complete collapse in the buckling mode.

(Confidential Abstract)

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INTRODUCTION

The fine continuous aluminum wires present in reinforced solid propellant (RFG) lead to a composite material with considerable load-bearing capacity. This has been demonstrated many times. These unique properties should make RFG a prime candidate for missile systems where exceptionally hostile environments must be withstood.

While investigating boost-sustain applications for RFG, it was discovered that some very thick-wall grains had developed circumferentially oriented interlaminar cracks. It was also found that subsequent low-temperature soaks would generally aggravate these cracks and sometimes cause the formation of new cracks. It became apparent that the potential of RFG would not be realized without solving the delamination problem.

Consequently, the main effort of this program was directed toward the solution of the delamination problem. Various approaches were investigated. These included an ambient-cure matrix and means by which radial reinforcement may be achieved. Other phases of the program involved the experimental determination of mechanical properties of RFG and the experimental examination of an externally pressurized thick-wall grain.

A balanced approach to reinforced propellant behavior was undertaken in this program. Several solutions to a basic problem concerning RFG were evaluated, some fundamental data essential to design were obtained, and the structural performance of a particular RFG design was investigated. More work in each of these areas is required. Obviously, the solution of the grain delamination problem is most important and most urgent.

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GRAIN FABRICATION

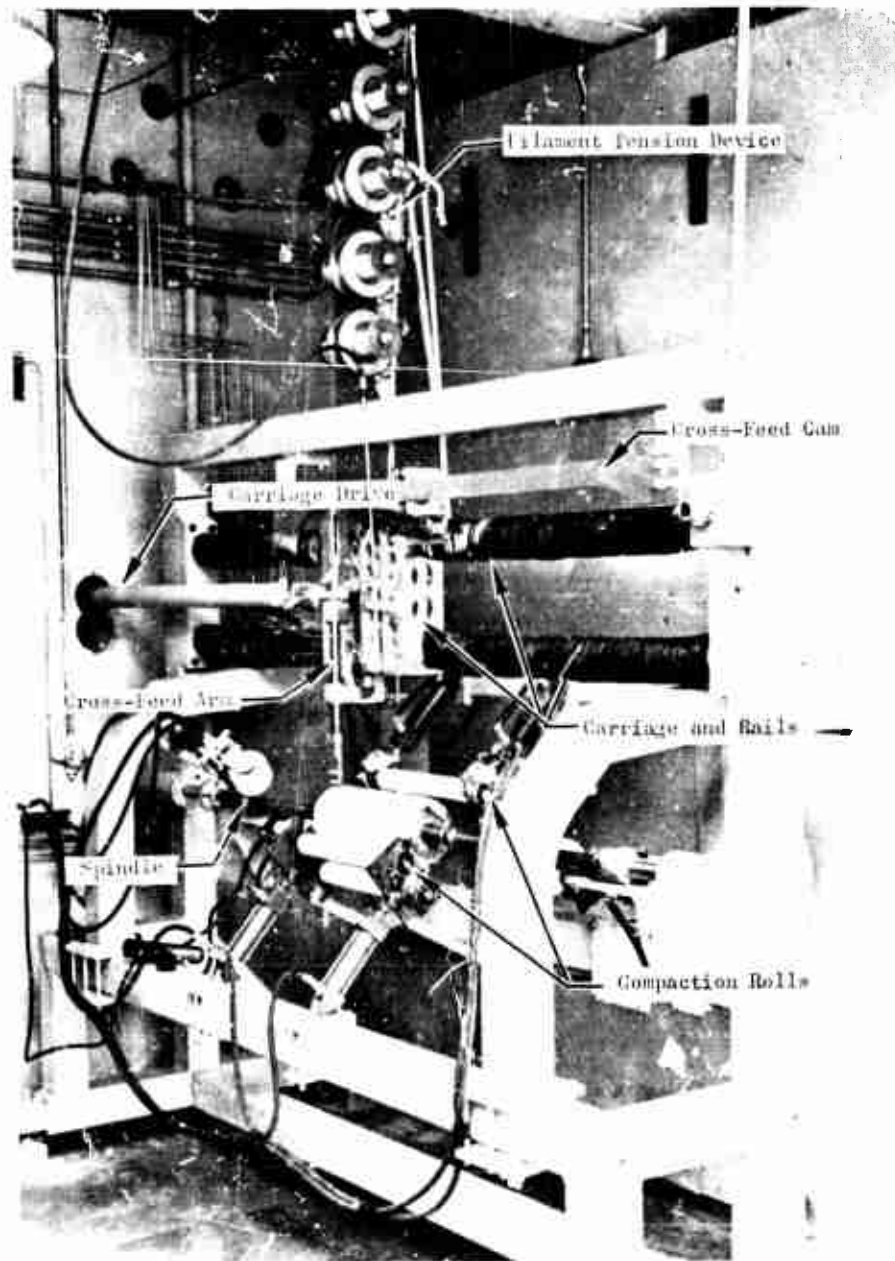
FILAMENT WINDING PROCESS

A filament winding machine capable of laying down precise winding patterns in accordance with predetermined winding paths is a prerequisite for fabricating reproducible composite test specimens. All of the filament reinforced grains required for the performance of this program were wound with such a machine, shown in Fig. 1.

Wire filaments are laid down on geodesic paths to eliminate wire slippage. Such calculated paths are achieved by a three-axis motion of the winding machine. Lateral movement is introduced by a cam-controlled carriage which travels parallel to the longitudinal axis of the mandrel, vertical movement is introduced by means of a cross-feed cam, and the spindle rotation bears a fixed relationship to the speed of the carriage. Four to ten wire filaments are laid down on the mandrel by a swiveling roller feed guide which aligns the filaments with each point of tangency of the grain surface. Minimizing the distance between the roller feed guide and the point of tangency on the grain reduces the unsupported length of the filaments and results in greater precision of wire placement.

A computer program is available to generate a description of any desired set of control cam. Geometric descriptions of the grains to be fabricated and sets of winding curve parameters are used as input data. After converting this information to punch card descriptions of the control cams that will produce the desired winding curves, and translating the data to automatic milling machine punched tapes, the cams are automatically cut. This is essentially equivalent to a tape-programmed winding machine, since a direct link is established between a mathematical statement of the mandrel shape and the desired winding curve to automatic operation of the winding machine.

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Figure 1. Cam-Controlled Winding Machine

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MATRIX COMPOSITION

Mechanical property test specimens fabricated for this program contain a common matrix. The choice is based upon several years of successful utilization in many reinforced propellant grains made for a variety of research purposes. The formulation consists of 13 weight-percent of carboxy-terminated polybutadiene binder and 87 weight-percent of a bimodal blend of ammonium perchlorate. Tris 1-(2 methyl) azirdinyl phosphine oxide, (MAPO) is added as the curative and usually amounts to approximately 2.5 weight-percent of the binder. This composition is similar in character to an uncured conventional composite propellant. However, the viscosity is higher than that used for making cast propellant grains. Solid propellant batch mixing processes are employed to produce the matrix feed for the filament winding process. Butarez Type I, a carboxy-terminated polybutadiene polymer is purchased from the Phillips Petroleum Company, and contains 1.23 weight-percent of active carboxyl groups. The formulation and mechanical properties of this matrix propellant are shown in Table 1.

TABLE 1

CHARACTERISTICS OF MATRIX PROPELLANT FOR FILAMENT-REINFORCED GRAINS

<u>Formulation</u>	
Ingredient	Weight Percent
Ammonium Perchlorate	87.000
Butarez CTL (Type 1)	12.675
MAPO Curative*	0.325
<u>Mechanical Properties** at 77 F</u>	
Tensile, psi	244
Elongation, percent	8.5
Modulus, psi	3500
*Tris 1-(2 methyl) azirdinyl phosphine oxide	
**Crosshead speed, 0.1 in./min	

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The oxidizer used to formulate the matrix propellant is prepared by grinding and blending two major particle size ranges of ammonium perchlorate. A ground portion of the regular grade is blended with the regular grade to the extent of 30 weight-percent and vacuum dried just prior to the mixing operation. The results of a micromerograph analysis showing the particle size and distribution curve for this bimodal blend is shown in Fig. 2, and is representative of the ammonium perchlorate used in the matrix composition. Because microscopic failure analyses of composites reveal that initial failure sites during tensile loading occur at the interfacial boundary between the larger particles and the binder, the maximum particle size of the ammonium perchlorate is maintained below 200 microns.

REINFORCING FILAMENT CHARACTERISTICS

Reinforcing filaments and strain gage wire were characterized under uniaxial tension conditions for a program concerned with the micromechanics of fiber-reinforced composites (Ref. 1). Several of these filaments were utilized in this program and include cold drawn 5056 aluminum alloy, high-strength steel, and Advance strain-gage wire. The performance of these materials is listed in Table 2. Experimental techniques and test conditions are described in detail in Ref. 1.

THIN-WALL GRAINS

The orthotropic nature of reinforced propellant was established by work performed during an earlier program on the response of this composite material. A continuation of this technical effort resulted in the determination of the magnitude of several orthotropic material constants for reinforced propellant (Ref. 2). During the current program, orthotropic material constant measurements have been extended to include aluminum wire reinforced composites wound at a 30-degree angle and high-strength steel wire reinforced propellant wound at a 54.7-degree angle. Such information is required for the efficient utilization of these composites as structural materials.

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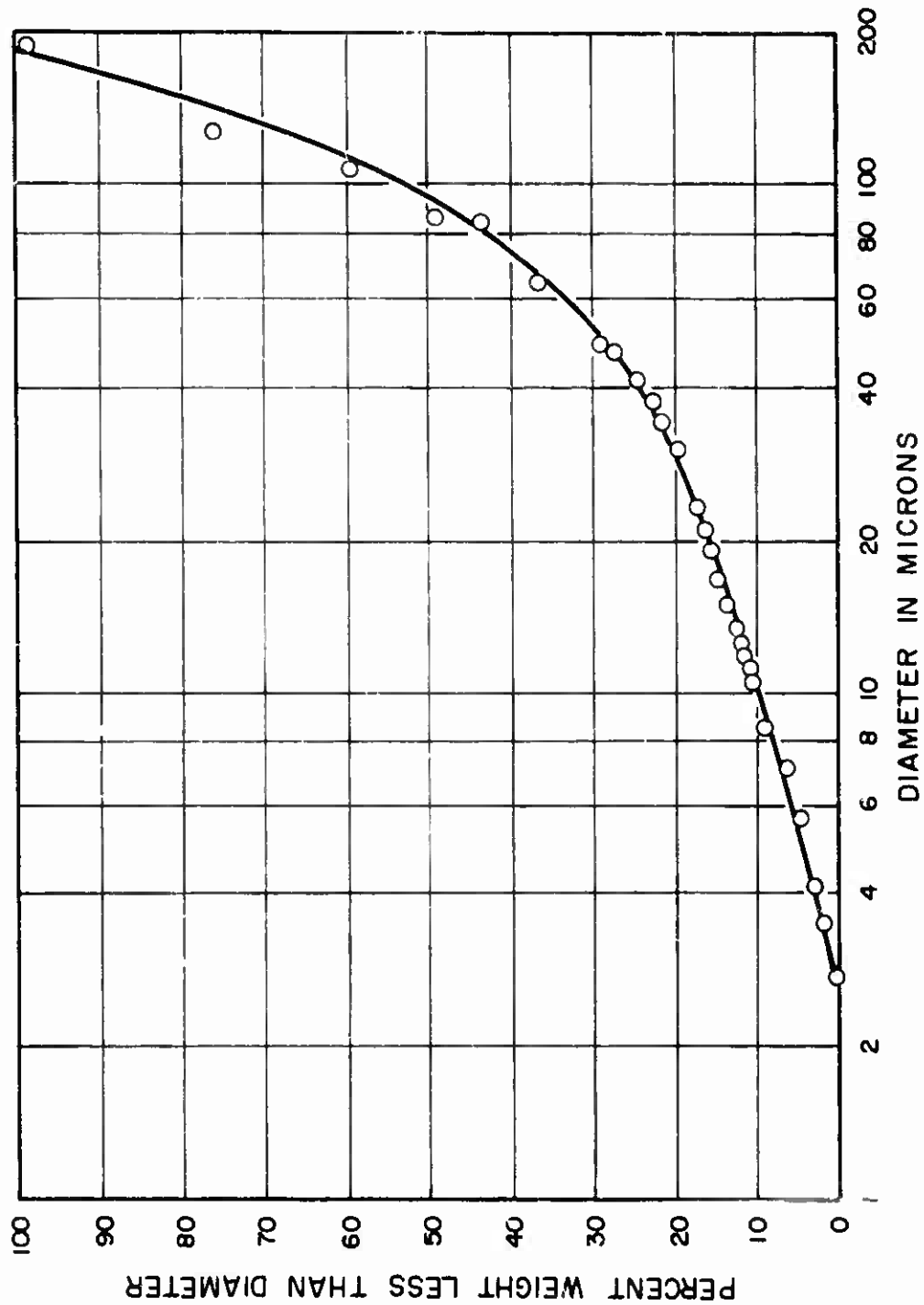


Figure 2. Ammonium Perchlorate Blend (70 percent coarse, 30 percent fine)

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TABLE 2

FILAMENT STRESS-STRAIN DATA

Filament Material	Diameter*, mils	Force, pounds	Stress**, $\text{psi} \times 10^{-3}$	Elongation, percent	Gage Length, inches	Initial Modulus, $\text{psi} \times 10^{-6}$
Cold Drawn Aluminum	4.98	0.2	10.3	0.11	10.5	9.41
		0.4	20.5	0.22		
		0.6	30.8	0.32		
		0.8	41.1	0.44		
		1.0	51.4	0.55		
		1.2	61.6	0.70		
		1.4	71.9	0.88		
		1.59	81.7	1.34		
High-Strength Steel	3.98	1	80	0.27	11.1	29.4
		2	161	0.55		
		3	241	0.82		
		4	322	1.10		
		5	402	1.40		
		7	563	2.44		
		7.03	565	2.50		
Advance Strain Gage Wire	3.90	0.2	16.7	0.06	11.4	27.9
		0.4	33.5	0.12		
		0.6	50.2	0.20		
		0.8	67.0	0.90		
		1.0	83.7	3.70		
		1.2	100.3	10.3		
		1.26	105.5	24.9		

*Average of five specimens

**Average of four or more tests, performed at a temperature of 70 ± 5 F, 0.2 in./min crosshead speed, 10-inch apparent gage length

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Four thin-wall cylinders were filament wound with live propellant matrix composed of 87 weight-percent of ammonium perchlorate and 13 weight-percent of polybutadiene binder. Three of these grains contained 5-mil-diameter, high-strength aluminum wire and one grain was fabricated with 4-mil-diameter, high-strength steel wire. In each case, the mandrel consisted of a 5-inch-diameter hollow aluminum cylinder, 25.5 inches long, with dome-shaped ends. The mandrel surface was covered with a thin coating of a silicone grease to permit easy grain removal and a single layer of fiber glass tape to prevent any contamination of the specimen by the silicone grease. Variation in grain diameter over the entire grain length was maintained within ± 0.020 inch for each specimen. This dimensional control was accomplished by frequent monitoring of the grain diameter, permitting machine corrections to be made as required. The above tolerance was important because the design wall thickness is only 0.125 inch. A minimum of wire slippage was obtained by carefully adjusting the wire tension to 150 grams for each filament, and maintaining temperature control of the matrix to minimize viscosity changes. All grains were cured in an oven maintained at a temperature of 170 F for 72 hours. This curing cycle is common to propellants formulated with carboxy-terminated polybutadiene and cured with MAPO.

Upon completing the curing cycle, each grain was cut into three cylindrical test specimens, 7 inches long. Cutting was accomplished with a high-speed (5000 to 10,000 rpm) milling cutter containing tungsten carbide teeth. The surfaces exposed with this wheel revealed clean smooth cuts and relatively undisturbed surfaces. Use of this technique minimizes the distance that cutting effects are transmitted into the test specimen where they may influence the test measurements.

A deviation from the usual procedure of pressing the end of the grain to remove it from the mandrel was instituted because of the thin-wall cross section which presents a very small area on which to exert force. Possible damage to the end of the specimens was eliminated by shrinking the mandrel surface away from the grains and manually slipping them off the mandrel.

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A small quantity of liquid nitrogen poured into the hollow center of the aluminum mandrel was sufficient to lower the mandrel temperature to approximately -20 F and release the wound grains. Specimen temperature was maintained above 32 F by the insulating character of the fiber glass tape separating the mandrel surface from the grain. Condensation of moisture on the grain surfaces was prevented by wrapping them with a polyester tape. Thus, mandrel removal did not subject the specimens to damaging conditions.

Compositions and geometries of the thin-wall grains, as well as a steel-staple reinforced grain and an aluminum screen wire-wound grain, are shown in Table 3. Fabrication of these special grains are described elsewhere in this report. Several thin-wall specimens are shown in Fig. 3.

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TABLE 3

WIRE-WOUND REINFORCED GRAINS

Grain No.	Grain Characteristics							Winding Angle, degrees
	Wire Type*	Composition, weight-percent			Dimensions, inches			
		Wire Content	Binder	AP**	ID	OD	Length	
041-23	Aluminum	9.55	11.76	78.69	5.03	5.31	25.45	54.7
041-25	Steel	24.10	9.87	66.03	5.04	5.35	25.45	54.7
041-26	Aluminum and Steel Staples	9.23 0.79	11.69	78.29	5.05	6.58	25.45	54.7
041-27	Aluminum***	7.17	15.79	76.04	5.04	5.27	25.45	54.7
041-30	Aluminum	12.54	11.35	76.11	5.03	5.29	25.45	30.0
041-36	Aluminum	9.32	11.79	78.89	5.04	5.32	25.45	54.7
041-37	Aluminum Screen	13.61	12.96	73.43	2.98	8.04	6.25	--

*Aluminum Wire--5056 alloy, 5-mil diameter

Steel Wire--High strength, brass coated, 4-mil diameter

Steel Staples--Standard commercial staples 1/2 by 1/4 inch

**AP--Ammonium perchlorate

***Ambient-cure grain-polyurethane binder

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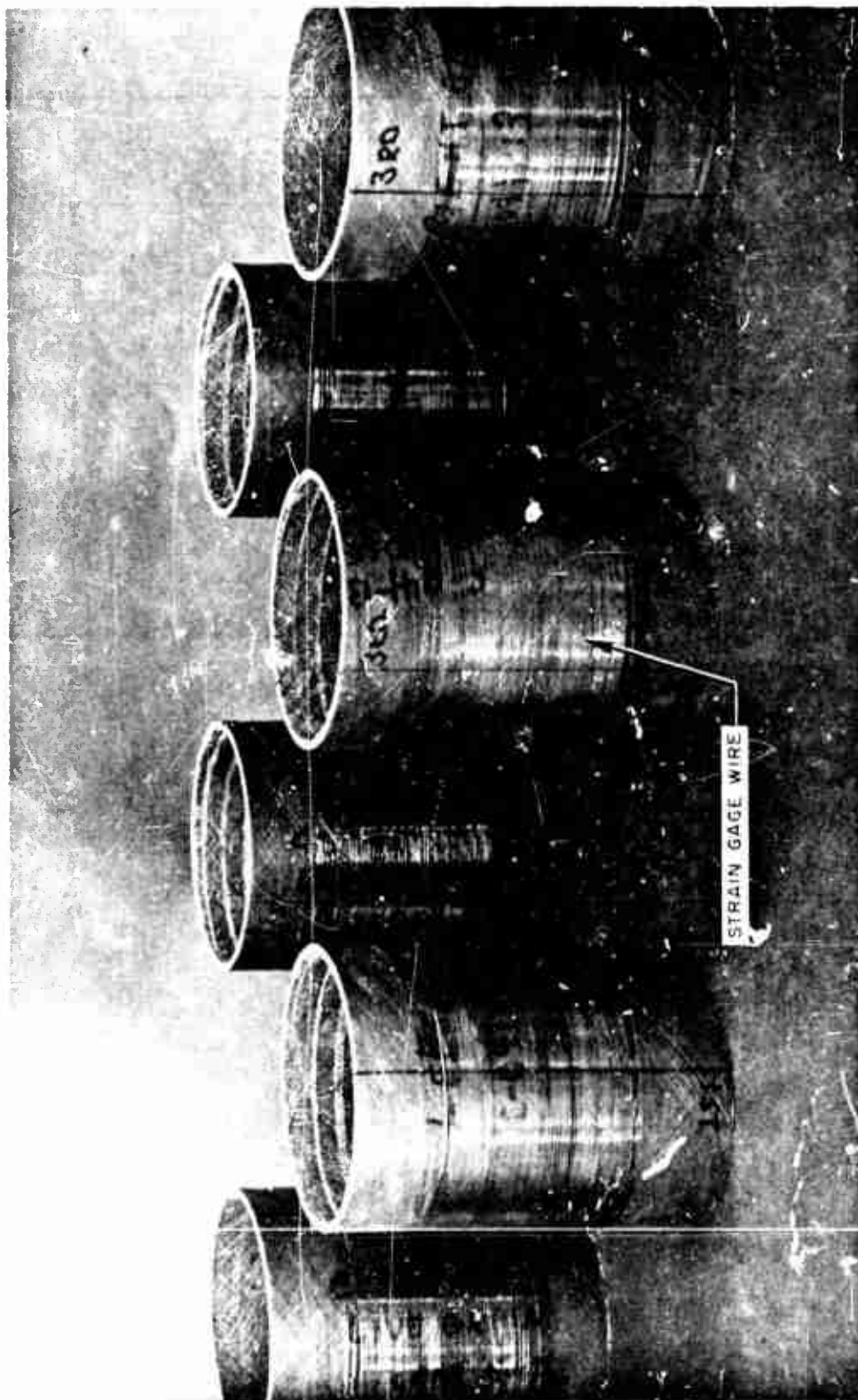


Figure 3. Thin Wall Specimens

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APPROACHES TO DELAMINATION PHENOMENA

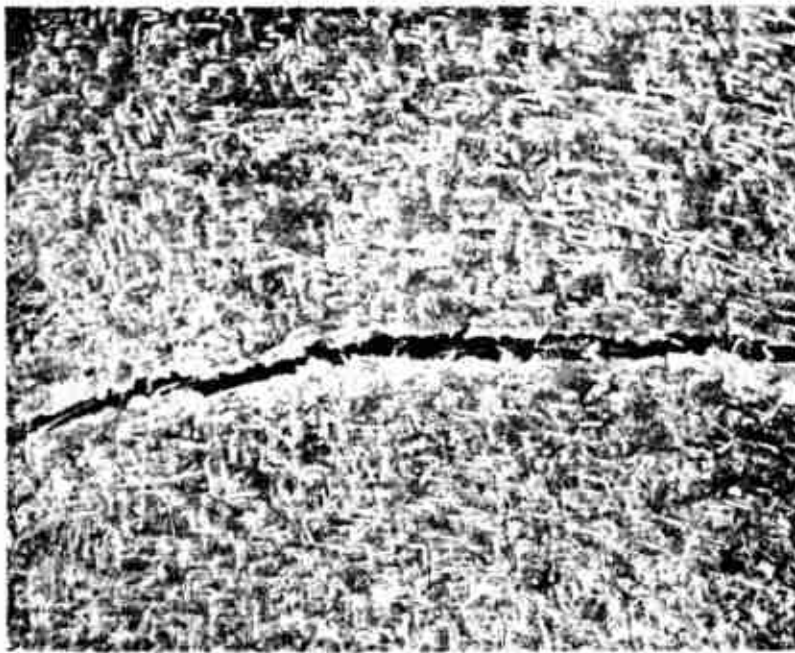
During the course of extending the concept of anisotropic burning of aluminum wire-reinforced grains to boost-sustain rocket motor applications, circumferential cracks were discovered in thick-web grains (Ref. 3). Many interlaminar flaws were observable in grains which had not yet been temperature cycled. Initially, longitudinal slots which are utilized in the boost-sustain design were suspected to be the cause of the cracks. However, further analysis and inspection of data and radiographs disclosed that the presence of slots did not appear to contribute to delamination. A photomicrograph of a circumferential crack in a sectioned thick-wall grain is shown in Fig. 4. In some cases, these flaws extend only a short distance into the grain, while in others they are continuous throughout the grain length. Thick-wall grains having initial interlaminar flaws, as well as those containing no apparent initial flaws, almost invariably displayed an aggravated crack condition when subjected to a cold-temperature cycle.

From this work it was concluded that these cracks could have been caused by one, or more probably, a combination of factors. The high OD to ID ratio of these grains is such that merely cooling from the cure temperature (170 F) to room temperature (72 F) imposes a strain distribution which exceeds the radial strain capacity of the grain. Process conditions which cause a residual strain in thick-wall grains at room temperature and grain design factors such as wire angle and matrix properties may contribute to the formation of flaws.

The major portion of this program has been devoted to preliminary investigations of techniques which would alleviate the formation of delaminated regions. The approaches considered include an ambient-cure grain (to reduce residual strains), increased matrix strength, and means by which radial reinforcement may be introduced.

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Figure 4 . Photomicrograph of Propellant Crack
in Filament Wound Grain

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AMBIENT-CURE GRAIN

A matrix system was developed under a company-sponsored program which has the desirable characteristics of high modulus and high strength coupled with the property of curing at room temperature. It was postulated that if it were possible (from a processing standpoint) to fabricate reinforced grains utilizing this matrix system, the delamination problem might be eliminated. The use of this matrix system would facilitate a reduction of the residual strains in a cured thick-wall grain. Also, the higher strength capability would also assist in the reduction of delaminations.

The first step to integrate an ambient-cure binder into reinforced propellant technology was taken in the form of fabricating a thin-wall grain. A 24.5-inch thin-wall cylinder was fabricated to ascertain processibility of the matrix system and for structural testing.

The ambient-cure matrix propellant contains a polyurethane binder made from commercially available materials. Polytetramethylene ether glycol (Polymeg 2010), an aliphatic diisocyanate (DDI 1410), and an isocyanated castor oil (Vorite 63) are mixed with the oxidizer to form the basic matrix for the winding process. The formulation contains 15 weight-percent of binder and 85 weight-percent of the same bimodal blend of ammonium perchlorate used with usual matrix propellant. At frequent intervals during the grain winding, small quantities of a solution of ferric acetyl acetonate in isodecyl pelargonate are added to the compaction roller which mixes this curing rate catalyst into the basic matrix and accelerates the ambient curing process. All other aspects of the grain fabrication and mandrel removal are identical to the process described for the thin-wall grains. The formulation and mechanical properties of the ambient-curing matrix propellant are listed in table 4. Compositions and physical characteristics of the ambient-cure grain are shown in Table 3.

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TABLE 4

CHARACTERISTICS OF MATRIX PROPELLANT FOR AMBIENT-CURE FILAMENT-REINFORCED GRAIN

Formulation

Ingredient	Weight Percent
Ammonium Perchlorate	85.000
Polymeg 2010	11.443
DDI 1410	2.370
Vorite 63	1.187
Isodecyl pelargonate solution of FeAA*	Trace

Mechanical Properties** at 70 F

Tensile, psi	252
Elongation, percent	4.1
Modulus, psi	18,300

*Curing catalyst

**Crosshead speed 2 in./min

In this manner the first ambient-cure reinforced grain was successfully fabricated. Subsequent torsion testing revealed that a less-than-optimum quantity of catalyst was applied during the winding operation and consequently a complete cure was not achieved. Funding limitations prevented studying means to achieve optimum amounts of catalyst. For the same reason, no fabrication of thick-wall grains was undertaken. This approach to the delamination problem is very promising and should be pursued.

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STAPLE-REINFORCED WIRE-WOUND GRAIN

Although there is general agreement that radial reinforcement would act as a deterrent to delamination, the method of simply and rapidly incorporating such reinforcing elements is not as clear. A means of accomplishing this was proposed by the ONR contract monitor. The unique idea consisted of manually introducing staples into the grain with a conventional staple machine. This appeared to provide a simple processing technique to evaluate the radial reinforcing concept.

Aluminum wire (5-mil) and the usual matrix propellant formulation shown in Table 1 were wound about a 5-inch-diameter mandrel to produce such a grain. Intermittently, the winding machine was stopped and standard steel staples were manually inserted in the grain at an angle of 54.7 degrees to the longitudinal axis. The chevron-like design shown in Fig. 5 was used. Eight equally spaced rows of radial staple reinforcing were introduced about the cylindrical grain surface in four discrete layers. Each layer is spaced 0.125 inch from the preceding layer with the first layer being introduced after filament winding the grain to a web thickness of 0.25 inch.

Grain fabrication proceeded in the usual fashion with no apparent difficulty. However, after completing the cure cycle and radiographing across the web, the legs of the steel staples were observed to have moved away from the radial direction and assumed positions parallel to the wound filament planes. Thus, all radial reinforcing characteristics were lost. Realignment of the steel staples is attributed to the rotary mixing action of the compaction roller. An X-ray photograph showing the extent to which the steel staples moved into the planes of the wire filaments is shown in Fig. 6. The composition of this grain is listed in Table 3.

Because of the staple motion indicated by the radiographs, no further tests were performed on this grain. The use of other staple reinforcing techniques which are more consistent with the existing helical winding fabrication process will be described subsequently.

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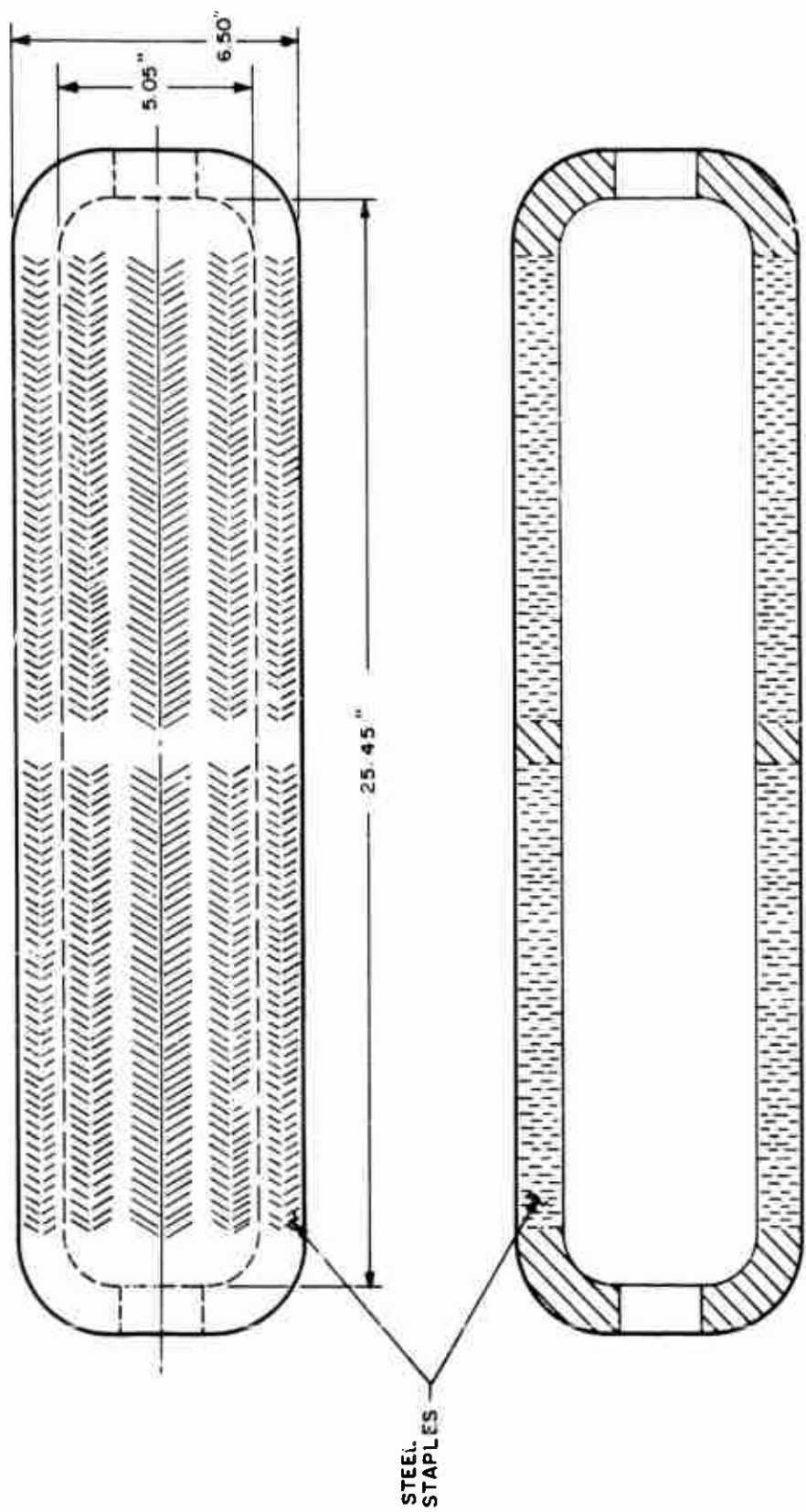


Figure 5. Staple Grain Design

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Figure 6. X-Ray of Radial Staple Reinforced Grain
(Diametric view shows staples aligned
in circumferential plane)

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SCREEN-WIRE GRAIN WITH RADIAL REINFORCING

Another means of making a delamination-free thick-wall grain was suggested by J. M. Crowley, ONR contract monitor. The proposed grain was to be a spirally wound aluminum screen wire grain. Therefore, a fabrication technique basically different from the helical winding process was employed. Aluminum screen-wire (5-mil) and matrix propellant wire were spirally wrapped about a 3-inch-diameter mandrel to form such a grain. The screen-wire was fed from a tensioned spool and the matrix was applied as needed at the nip angle between the compaction roller and the grain surface. After every five revolutions of the mandrel, the winding was stopped and a screen puncturing device was rolled over the several layers of propellant, thus breaking a limited number of aluminum wire filaments and orientating them in a radial direction. By repeating this process throughout the grain fabrication, radial reinforcement was achieved over the entire web. A schematic of the machine used for winding the screen-wire grain is shown in Fig. 7.

Matrix propellant for this screen-wire reinforced grain was especially formulated to suit the screen winding process. The matrix differs from that used to fabricate the mechanical property test specimens in that it contains 2 weight-percent less of the bimodal blend of ammonium perchlorate. Thus, a reduction in the matrix propellant viscosity is obtained and the matrix can more readily penetrate the small openings in the screen-wire. The formulation is composed of 15 weight-percent of carboxy-terminated polybutadiene binder and 85 weight-percent of ammonium perchlorate. As in the case of the usual matrix formulation, MAPD is used for crosslinking and batch mixing is employed to prepare the mixture for grain fabrication. The standard curing cycle developed for this polymer system was utilized, and consists of holding the grain in a 170 F oven for 72 hours. The matrix formulation for the screen-wire reinforced grain is given in Table 5.

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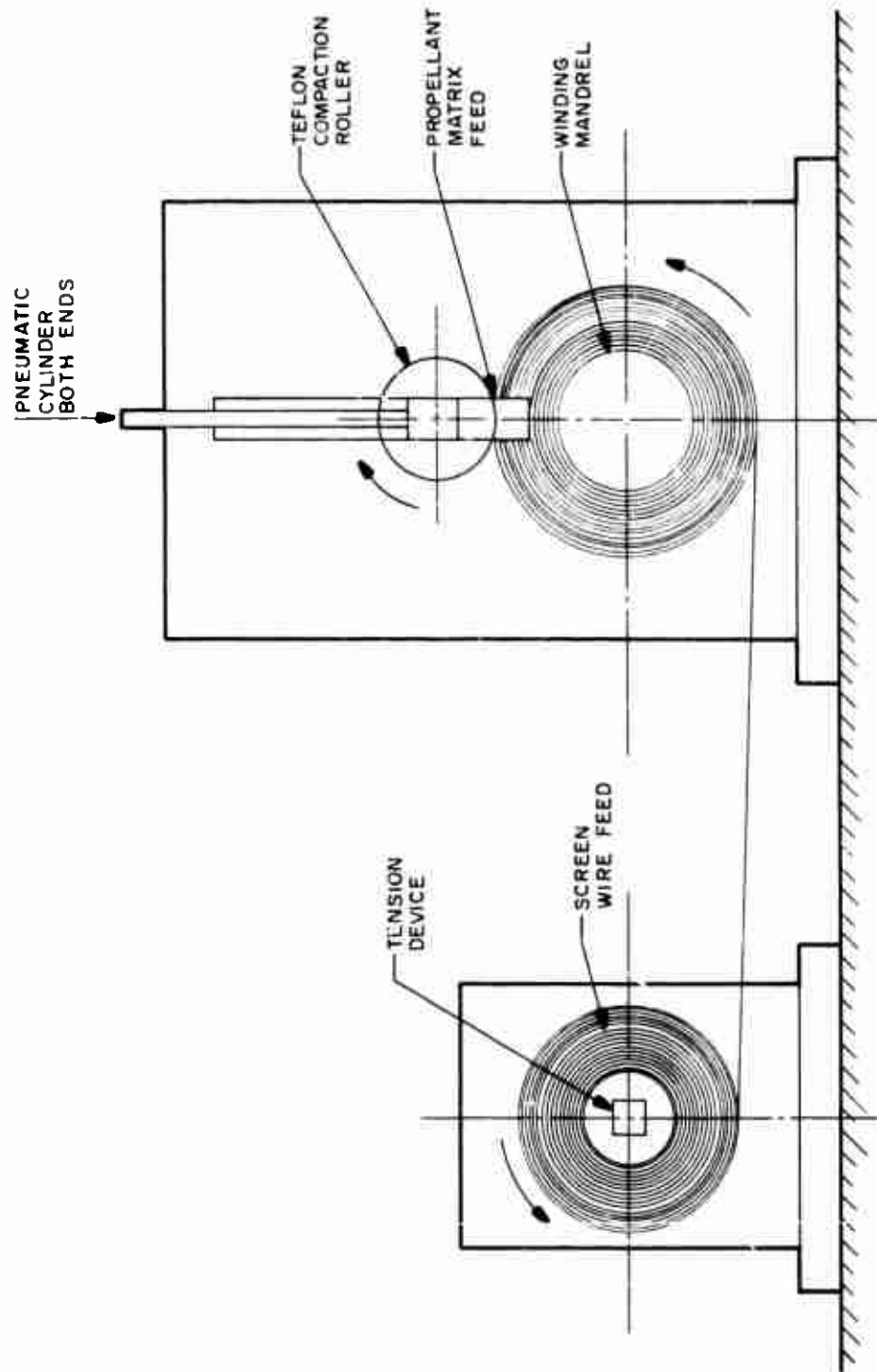


Figure 7. Schematic of Machine for Winding Screen-Wire Grain

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TABLE 5

FORMULATION OF MATRIX PROPELLANT FOR SCREEN-WIRE REINFORCED GRAIN

Ingredient	Weight Percent
Ammonium Perchlorate	85.000
Butarez CTL (Type 1)	14.625
MAPO Curative*	0.375

*Tris 1-(2 methyl) azirdinyl phosphine oxide

After curing, both ends of the screen-wire grain were machined with a high-speed tungsten-carbide-tipped cutter to provide a finished grain length of 6.25 inches. The normal procedure of pressing the end of the grain to slip it off the mandrel was used, because the 8-inch OD presented a large cross-sectional area upon which to exert a small force with little danger of damaging the specimen. Press force was transmitted to the grain surface through a steel plate covering the entire cross section of the grain. After initial movement of the grain, it was manually slipped off the untapered mandrel and placed in an air-conditioned room to await physical measurements and temperature cycling.

A visual inspection of the screen-wire grain was made approximately 24 hours later and preliminary to making physical measurements. A delamination in one end of the grain was discovered at this time. This flaw was located very close to the mid-point of the web cross section and measured 1.26 inches from tip to tip. Visual examination performed 3 days later indicated that the crack increased in length to 1.5 inches and became visible on the opposite end of the grain. Measurements of the ID and OD were taken at eight positions on each end of the grain immediately following discovery of the delamination and again after the flaw became visible at the opposite end. These dimensions showed no significant changes within the ± 0.020 -inch accuracy of the measurements. The location of the delamination is shown in Fig. 8 and 9.

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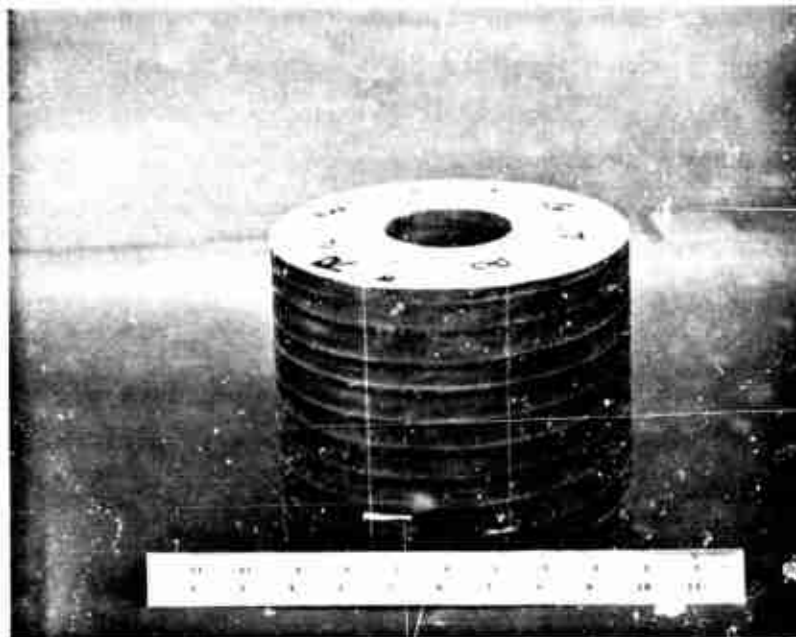


Figure 8. Reinforced Grain No. 041-37 Wound with Aluminum Screen Wire

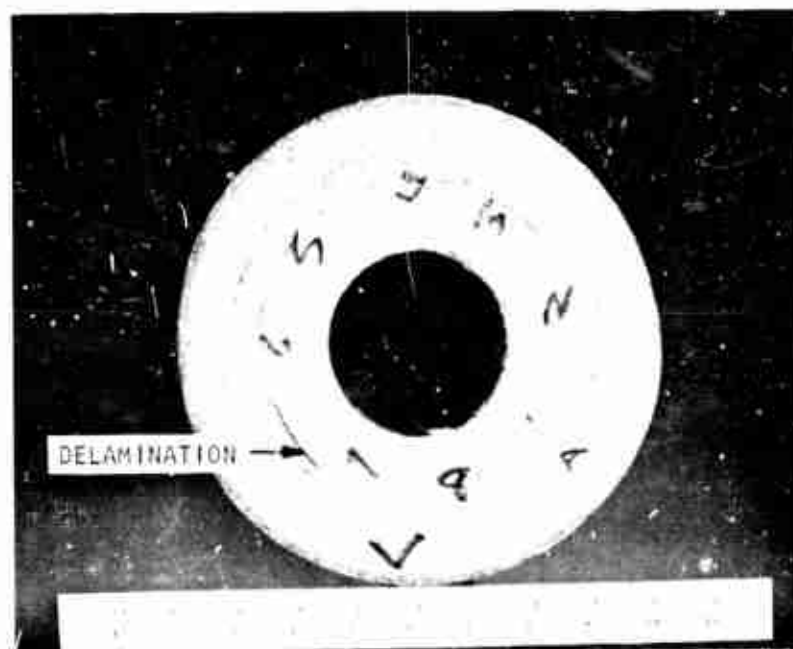


Figure 9. End View of Grain No. 041-37

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Subsequent to the visual inspection, radiographs of the entire screen-wire grain were taken tangent to the cylindrical surfaces. One major delaminated area was present which extended over the full grain length. Nearby, a second flaw appeared close to one end of the grain. However, this crack was relatively short and was probably associated with the major defect. These delaminations appeared as straight lines in the X-ray shown in Fig. 10.

Various reasons for these cracks may be advanced, such as those connected with grain fabrication. For instance, the lack of sufficient matrix in a particular area would conceivably result in a crack or, in this case, a lack of sufficient radial reinforcement in a specific area. It is possible that filament or wire spacing is an important parameter, particularly in the radial direction. This may be illustrated in the following manner. If screen-wire or wire filaments are brought together so that they contact or almost contact each other, obviously little matrix can be present to assume the strains which develop. Some research in this direction might prove useful and help to explain the circumferential cracks such as shown by the photomicrographs of Fig. 4 and 11. These are 5X enlargements of delaminations in a filament-wound grain and in the screen-wire grain. In general, these flaws are similar, although the edges of the filament-wound grain crack are much rougher and appear to move through different circumferential planes. Such a difference can be anticipated in view of the interwoven character of the filament-wound grain relative to the screen-wire-wrapped laminate.

OTHER APPROACHES TO THE DELAMINATION PROBLEM

Improving wire-reinforced propellant processing techniques to minimize residual stresses within the grain may offer a solution. Currently, wire-reinforced propellant utilizes a polybutadiene type of matrix binder requiring a 3-day curing cycle at elevated temperatures. Assuming a uniform curing reaction throughout the grain, a condition of minimum

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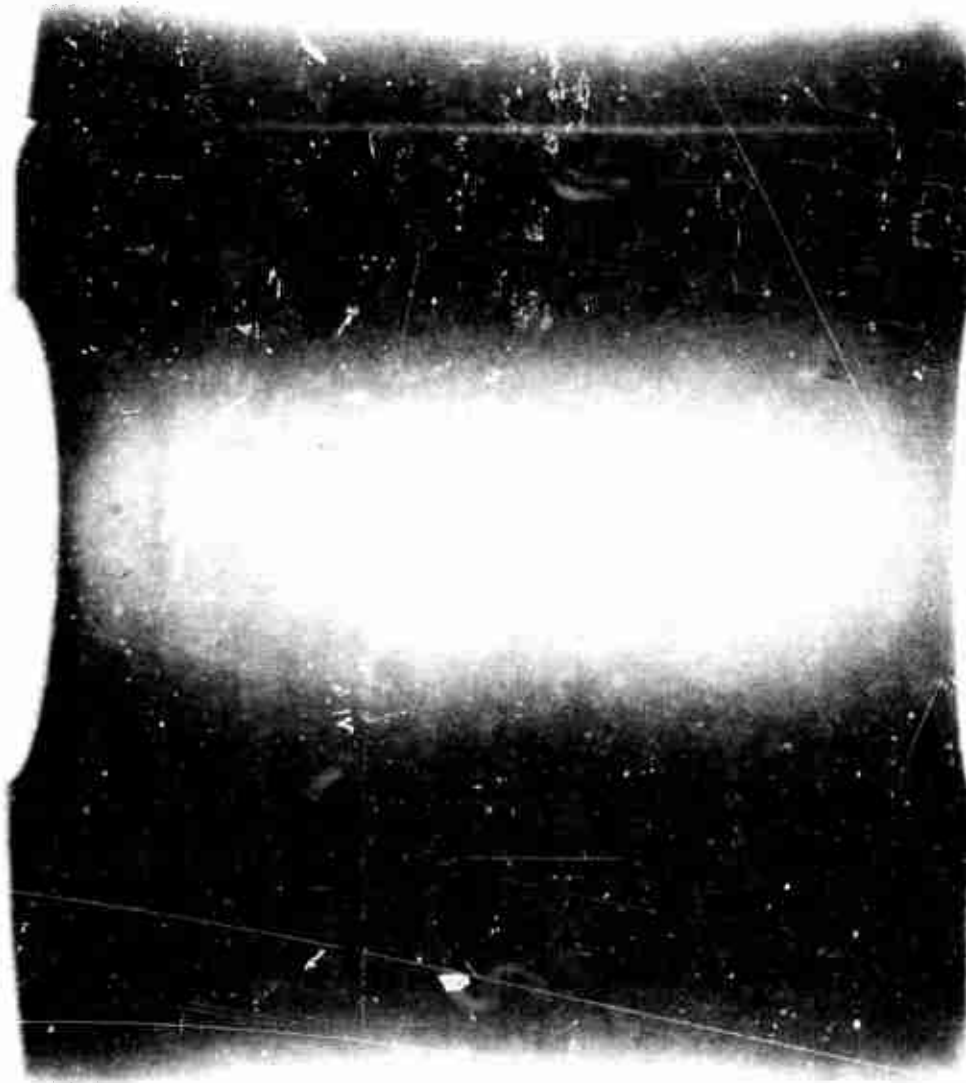
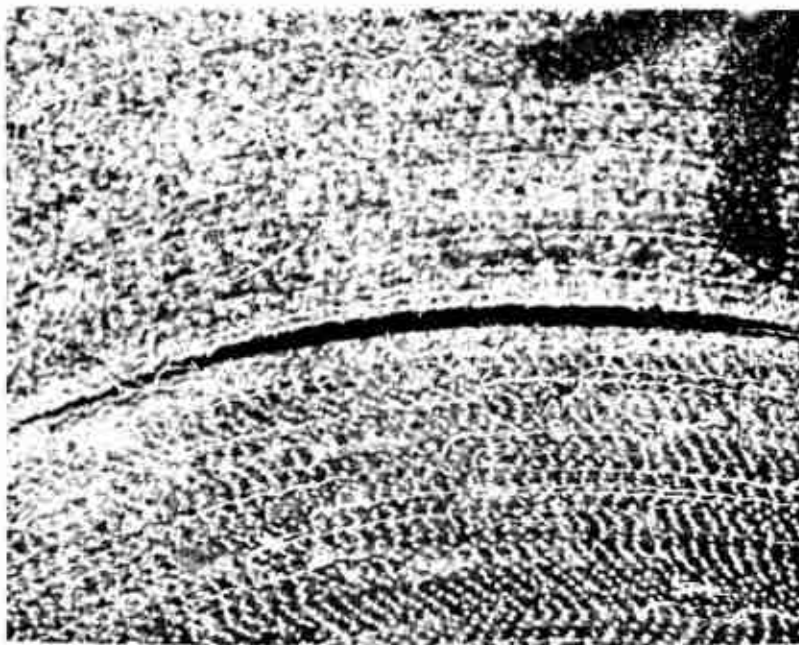


Figure 10. X-Ray of Aluminum Screen-Wire Grain
Showing Delamination

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5X

Figure 11. Photomicrograph of Propellant Delamination
in Screen-Wire-Reinforced Grain

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stress exists within the structure at the curing temperatures. However, thermal stresses are introduced when the wire-reinforced grain is removed from this environment and subjected to the usual ambient temperature conditions. These stresses are increased further when the reinforced propellant temperature is lowered (-70 F) during temperature cycling.

The preceding explanation is an over-simplification because the curing reaction is not uniform throughout the composite. Wire-reinforced grains are cured on a winding mandrel which can conduct heat to the ID, although most of the heat enters the grain from the exposed outer surfaces. Thus, the cure proceeds in general from the outside to the inside and can produce complex stress conditions within the structure.

Utilization of a reinforced propellant matrix capable of ambient curing on the mandrel as the grain is being wound provides a unique improvement in the process which can alleviate thermal stresses resulting from the usual curing cycle. This concept may be reduced to practice by making a matrix premix having the correct formulation except for a reaction or curing catalyst which is added via the compaction roller during the grain fabrication. The catalyst mixes with the matrix propellant on the surface of the grain and curing starts as the reinforcing wires are being wound into the matrix. Curing rates can be altered by varying the amount and concentration of the catalyst applied to the rolls, and adjusted to match the fabrication rate. The successful fabrication of a thin-wall grain with this matrix system has been previously described. The true test of this approach requires the fabrication of a thick-wall, ambient-cure grain. In addition to reducing detrimental residual stress fields, this process may facilitate radial compressive prestressing (as in the autofrettage technique). This may further extend the low-temperature capability of thick-wall grains.

Minimizing internal stresses in wire-reinforced propellant may be achieved in another manner. Many continuous filament-wound reinforced grains have been fabricated by the Research Division utilizing carboxy-terminated

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polybutadiene matrix propellants and static fired to evaluate the ballistic characteristics of the aluminum wire-reinforced system. Delaminations were not apparent in these grains. However, the web-thickness was only 0.5 inch because this is sufficient to attain a pressure and thrust equilibrium needed for obtaining ballistic data. Delaminations were first observed in thick-wall grains having OD/ID ratios of 2.5 or greater. Because 0.5-inch thick-walls can be satisfactorily processed with no evidence of cracking, incremental winding and curing may be used as a process technique for fabricating thick-wall grains. Although this approach introduces the possibility of interfaces between increments, this may not be serious because wire-reinforced restrictors have been wound about cured grains without difficulty.

Another method for solving the grain delamination problem is to increase the radial strength of wire-reinforced propellant by distributing the stresses more uniformly throughout the thick-web rather than attempting to relieve existing stresses. An increase in the radial strength of wire-reinforced propellant can be achieved by incorporating short aluminum wire filaments into the grain so that a radial orientation and reinforcement at the local level is obtained. Such an effect can result from feeding a quantity of short filaments along with each increment of matrix propellant. A portion of these short fibers are given a radial orientation by the action of the continuous wire filaments working their way into the surface of the matrix. This technique is compatible with the helical winding process and will achieve radial reinforcement. Consideration should also be given to employing short segments of aluminum wire in combination with the ambient-cure matrix system.

Developing a matrix binder more suitable for use with wire-reinforced propellants is another approach that may eliminate delaminations. A carboxy-terminated polybutadiene binder was adopted during the early research on reinforced propellant. Originally, this matrix was chosen for ease of processing and good ballistic performance. Until the appearance of cracks in thick-wall grains, it appeared that the Butarez resin

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used for cast propellants was adequate for wire-reinforced propellants. It now appears that a tougher, stronger matrix may be required because the most effective use of RFG will involve its ability to withstand severe environments. Where structural loads are to be carried by the propellant grain, the propellant undoubtedly will be improved if a high-strength binder is selected to support the high-modulus reinforcing wire.

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MATERIAL PROPERTIES TESTS

The primary aim of this phase of the program was to investigate the orthotropic characteristics of RFG. This information is essential in predicting the response of RFG to mechanical and thermal loadings. The test series described below is an extension of the work performed under an earlier task of the current contract and was reported in Ref. 2.

Because orthotropic properties can be very sensitive to changes in wire angle and modulus, the test series was designed to obtain an estimate of the effects of these parameters. The original plan included two additional winding angles (one higher and one lower than the 54.7-degree angle previously considered and one wire type with a higher modulus than aluminum (steel). Redirection of the main program effort to the delamination problem resulted in the elimination of part of this plan.

Thin-wall cylindrical specimens were used for both uniaxial and torsion tests. Fabrication and composition of the specimens have been described in a previous section.

INSTRUMENTATION AND RECORDING

Recording

All the measuring devices used during the test series were connected to individual galvanometers which produce traces on an oscillograph. Calibrations (which will be described separately below) consisted of correlating oscillograph trace displacements with known increments of the quantities being measured. This data retrieval system enabled the simultaneous, continuous monitoring of all the parameters of interest.

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Instrumentation

Longitudinal Displacement. Clip-type transducers, fabricated from stainless-steel sheets, 0.015 inch thick, were used to measure longitudinal displacements of the cylindrical specimens. Two 350-ohm foil strain gages were applied to the central portion of the transducer. A change in distance between the legs of the transducer results in bending of the central portion. One strain gage is in tension and one is in compression. Because the gages were wired as adjacent legs of a Wheatstone bridge, the output of the two gages is added. The bridge was excited by a d-c electrical source.

Calibration of the displacement transducers was accomplished by attaching the transducer legs to two blocks of plastic firmly fixed on the anvils of a vernier caliper. Increments of displacement were then applied by the fine adjustment screw of the caliper and the output of the gage bridge was recorded on the oscillograph.

The attachment of the grain specimens was achieved by adhering small pieces of plastic to the grain surface with Epon 820. The feet of the transducers were then screwed to the plastic tables. This method of attaching the transducers to the grain is the same as that used in the calibration setup.

Circumferential Strain. The circumferential strains were obtained by means of Constantan strain gage wire helically wound in a very close pitch (32 turns per inch) on the grain surface. The wire was wound into a 5- to 10-mil layer of Epon 820 and cured. After curing, the wire was cut at two places and electrical leads were attached. This effectively resulted in a hoop gage which averages the strains over its entire length. For all the thin wall specimens tested, two such hoop gages were utilized in the central 2 inches of the grain. The initial resistance of each of the gages was approximately 130 ohms.

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The quantity actually measured was the resistance change of the known initial resistance. Because the Advance strain gage wire has a gage factor of approximately 2 (even after the wire passes its yield point), the hoop strain is easily calculated from the relation

$$E_e = \frac{\Delta R}{\text{Gage factor} \times R_i}$$

The amount of oscillograph trace deflection corresponding to a given resistance change was obtained by inserting a resistance decade box in series with the gage and adding increments of resistance.

As a check on the use of hoop wound gages to measure circumferential strain, a clip-type displacement transducer was used to determine grain diameter change during selected tests. This transducer was calibrated in the same manner as the longitudinal transducers. There was close agreement in the circumferential strains determined in this manner and those obtained from the hoop gages during the tests where both methods were used. A high confidence measure can therefore be assigned to the hoop gage data.

Internal Pressure. Internal pressure measurement was obtained by means of a Teledyne pressure transducer. Calibration was performed by means of a standard dead weight tester. Internal vacuum measurement was obtained by means of a Statam differential pressure transducer. A vacuum pump was used to achieve a vacuum equivalent to 28 inches of mercury. A mercury manometer was plumbed into the vacuum line and calibration of the transducer was accomplished by drawing vacuum in increments of inches of mercury and recording the corresponding oscillograph trace displacement.

Axial Load. Axial loads were applied by an Instron test machine and measured by an Instron load cell. Calibration of the load cell was accomplished by a Morehouse proving ring.

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Angular Rotation. A specially designed transducer was fabricated to measure relative rotation of two cross-sections of a twisted specimen. This transducer consisted of a cantilever beam which is attached to one cylinder section by means of a piece of plastic bonded to the surface. Two strain gages bonded to the beam produce an electrical signal when the beam is deflected. A cylindrical peg was similarly affixed to the cylinder at another cross-section. As the two sections were rotated with respect to each other, the beam was deflected and a signal was generated by the strain gages. Calibration of the transducer involved a correlation of trace displacement with imposed known increments of relative angular displacement.

TEST SET-UP AND PROCEDURES

Each of the thin-wall grains was glued with Epon 820 and curing agent T into aluminum end fixtures (Fig. 12). Considerable care was employed in the alignment of the grain in the end fixtures. All gluing operations were conducted on a surface plate and a right angle square was used to set the generators of the cylindrical grain normal to the end fixtures. An assembled, fully instrumented test specimen is shown in Fig. 12.

Compression Tests

To ensure axially of the applied compressive loads, a shallow hole was countersunk in the center of the top end fixtures. A steel ball was placed in this hole, and the assembly was set in the Instron test machine with the steel ball along the axis of the compression cell. The compressive load was applied slowly (crosshead speed of 0.05 in./min) to the desired load level. One of the specimens was loaded in this manner until a maximum load was reached. After achieving this maximum load, the crosshead motion was reversed and the load on the specimen was relieved.

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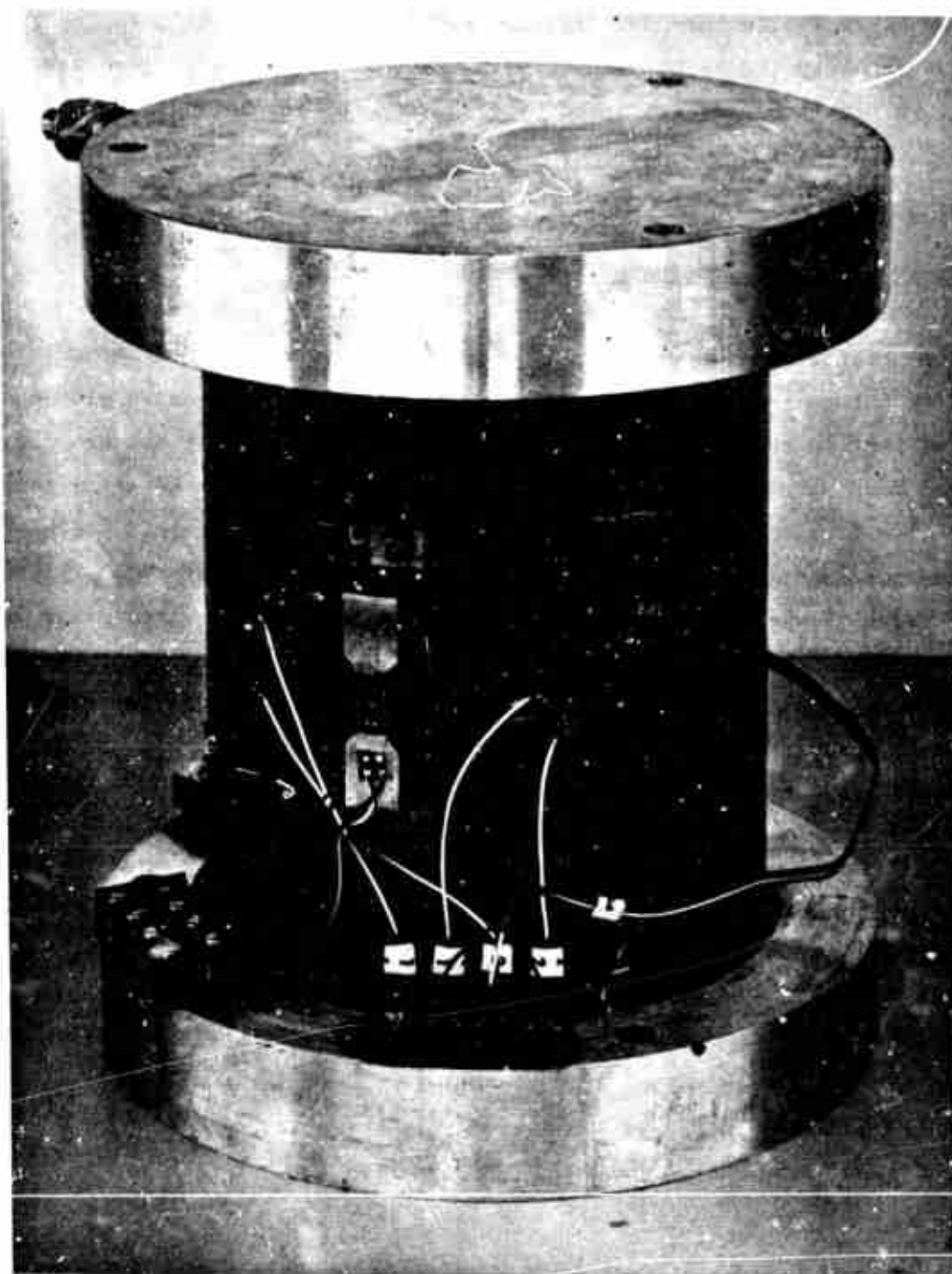


Figure 12. Instrumented Thin Wall Test Specimen

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Tension Tests

Two plates containing centrally located shafts were bolted to the end fixtures. A steel block was screwed onto each of the shafts. The steel blocks were fitted into the Instron tensile grips, and the axial tensile loads were applied by pulling on these blocks. The tensile load was applied slowly (crosshead speed of 0.05 in./min) to the desired load level.

Hoop Tension and Compression Test

Stress states of hoop tension and compression are obtained by means of combining internal pressure and internal vacuum with axial compression and axial tension respectively. Because internal pressure (or vacuum) causes a 2:1 hoop stress to axial stress in thin-wall cylinders, an axial compression (or tension) must be applied to cancel the axial stress. Gaseous nitrogen was used for internal pressurization and a vacuum pump was used to create vacuum. The balancing of the axial stress with axial load was incremental. The sequence consisted of the application of an appropriate increment of axial load. Although this procedure is not as satisfactory as continuous cancellation of the axial stress, sufficiently good data were obtained by this technique.

Biaxial Stress Tests

Various ratios of axial to circumferential stress can easily be obtained by means of adding to (or subtracting) axial stress to the 1:2 ratio obtained from internal pressure or vacuum loadings. Three thin-wall specimens were tested in this manner.

Torsion Tests

A special torsion fixture was designed for use in conjunction with an Instron test machine. This fixture is bolted to the crosshead of the machine and

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torque is applied to the specimen by means of a cable around a pulley as shown in Fig. 13. The cable is connected directly to a tension load cell. The torsion fixture was so designed that the same specimen configuration and end plates could be used as were employed for the other material properties tests. The cylinder shown in the fixture (Fig. 13) is a split cylinder used in calibrating the angle-of-twist transducer.

The test procedure consisted of applying increasing torque at a constant rate to a certain maximum and then unloading. The maximum torque applied was governed by the capacity of the torsion fixture.

RESULTS

The maximum shear stress level imposed on each specimen during torsion testing was governed by the capacity of the torsion fixture. In each case the torque-shear strain curves were straight lines. The maximum shear stresses applied were not sufficiently great to cause yielding in any specimen. The torsional moduli for the specimens tested are presented in Table 6.

TABLE 6

TORSIONAL MODULI FOR TESTED SPECIMENS

Grain Number	Winding Angle, degrees	Wire Type	Torsional Modulus, psi
041-25	55	Steel	295,900
041-23	55	Aluminum	152,800
041-30	30	Aluminum	127,200

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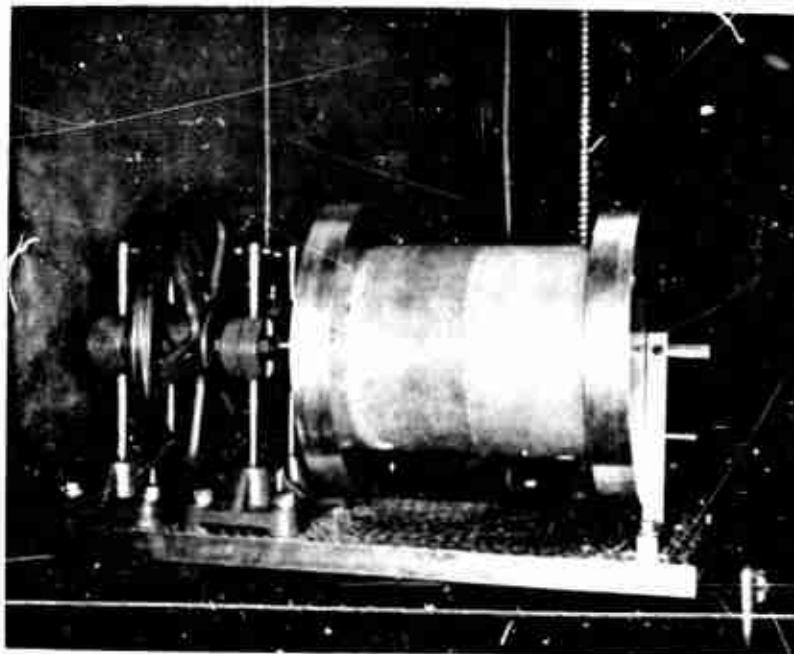


Figure 13. Torsion Test Fixture

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Other aspects of torsional behavior were observed. For example, a 55-degree aluminum wire specimen displayed no appreciable relaxation from a 670-psi shear stress level over a 5-minute period. Also, each of the specimens were subjected to from 10 to 15 load cycles with no apparent response changes. The reason for this behavior lies in the closeness of the winding angles to 45 degrees, the angle where the maximum direct stresses occur under torsion. Consequently, the wires are predominating the response in the ranges tested.

An ambient-cure, thin-wall grain was also tested in torsion. It became apparent that a complete cure was not attained, and the test was terminated.

Two specimens each of 54.7-degree steel wire grain and the 30-degree aluminum wire grain were subjected to hoop and longitudinal tension. The stress-strain curves characteristically displayed a linear initial portion (similar to those presented in Ref. 2). The steel wire specimens did not exhibit any appreciable relaxation or creep behavior. However, there was considerable creep associated with the hoop behavior of the 30-degree aluminum wire grain. This is reasonable because the lower winding angle (compared with the 54.7-degree winding angle) would result in hoop behavior that more strongly reflects the viscoelastic properties of the matrix. Creeping of the grain in the hoop direction caused difficulties in incrementally cancelling the pressure-induced axial stresses. The properties determined by these tests are presented in Table 7.

TABLE 7

GRAIN PROPERTIES

Grain	E_z , psi	ν_{ze}	E_e , psi	ν_{ez}
55-degree Steel	52,600	0.415	171,000	2.54
30-degree Aluminum	119,000	2.97	10,800	0.17

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It should be emphasized that the results presented represent the results of single tests. Therefore, no estimate of scatter can be advanced. Any subsequent related effort should include several tests at specific tests conditions to establish confidence limits.

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PERFORATED CORE GRAIN

Thick-wall reinforced propellant should be capable of withstanding high external pressure loading and therefore appears promising for certain deep submergence applications. For these applications, there is a necessity for high-volume loading of propellant, which thereby limits the size of the grain perforation. Special designs must be selected to provide the high mass flowrates necessary to attain high thrust-to-weight ratios usually required. A new approach to the design of such a grain was conceived on a company-sponsored program and forms the basis for the current work.

The design concept consists of a reinforced-core grain containing a prescribed pattern of radial holes which is overwrapped with a shell of reinforced propellant. This design preserves most of the structural strength of the core and provides the added burning surface required for high mass flowrates. Various patterns of radial perforations and depths of holes provide versatility to this design approach. The propellant grain fabricated for testing during this program had a core wound at 70 degrees and an outer propellant shell wound at 54.7 degrees. Details of the fabrication of this grain are presented in Ref. 2. A schematic of this grain is shown in Fig. 14 and the completed grain is shown in Fig. 15. The completed grain was then prepared for testing by external pressurization.

TEST SET-UP AND PROCEDURE

A chamber, consisting of a 6-inch, stainless-steel shell with heavy welded flanges, was modified to accommodate and apply external pressure to the perforated core grain. One flange was machined to achieve an O-ring seal with one end fixture of the grain. Because pressure was to be applied by hydraulic fluid, a thin protective coating of PRC synthetic rubber was applied to the grain surface.

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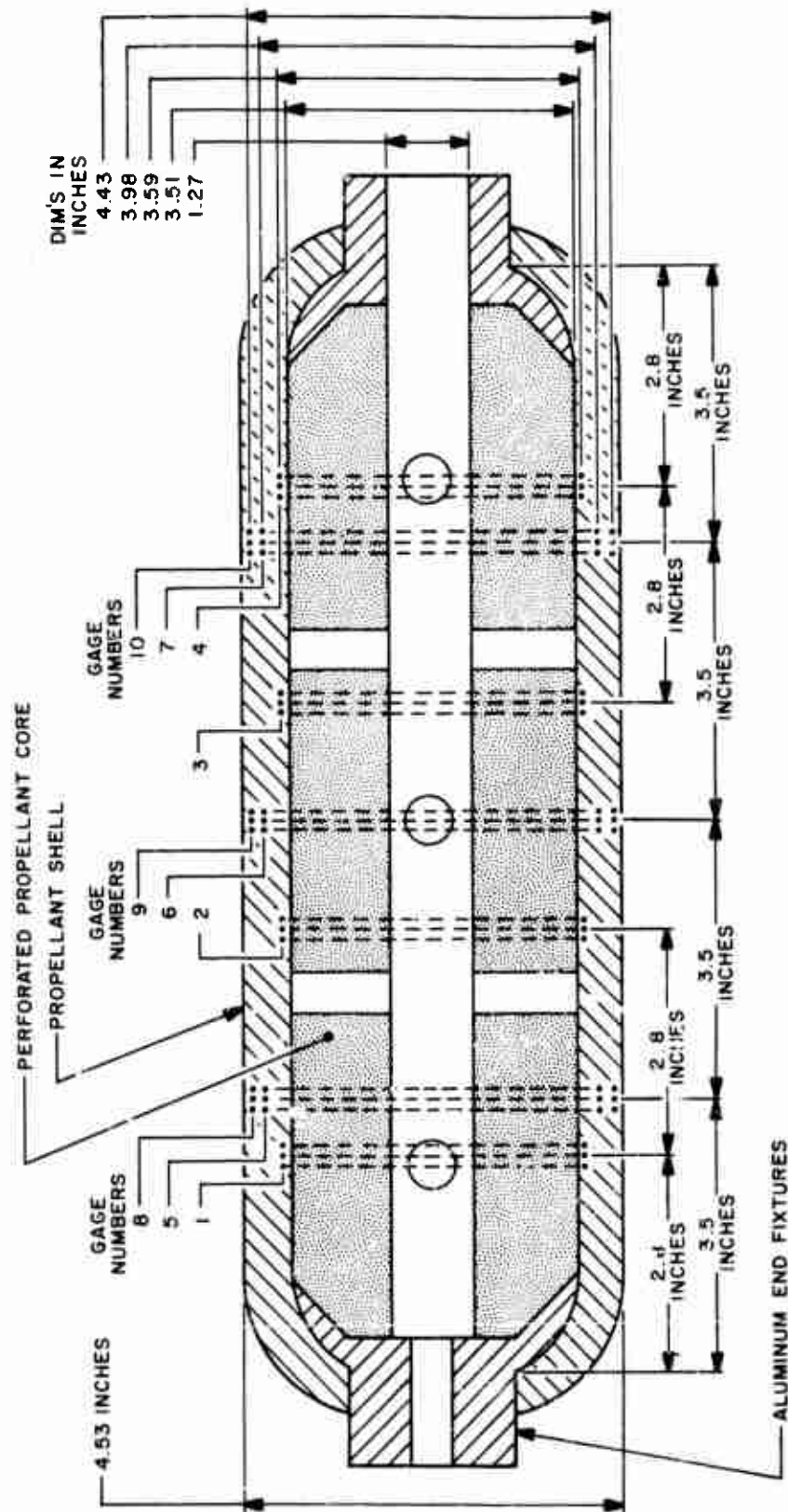


Figure 14. Grain No. 041-17 (perforated core plus external shell) for External Pressurization Tests

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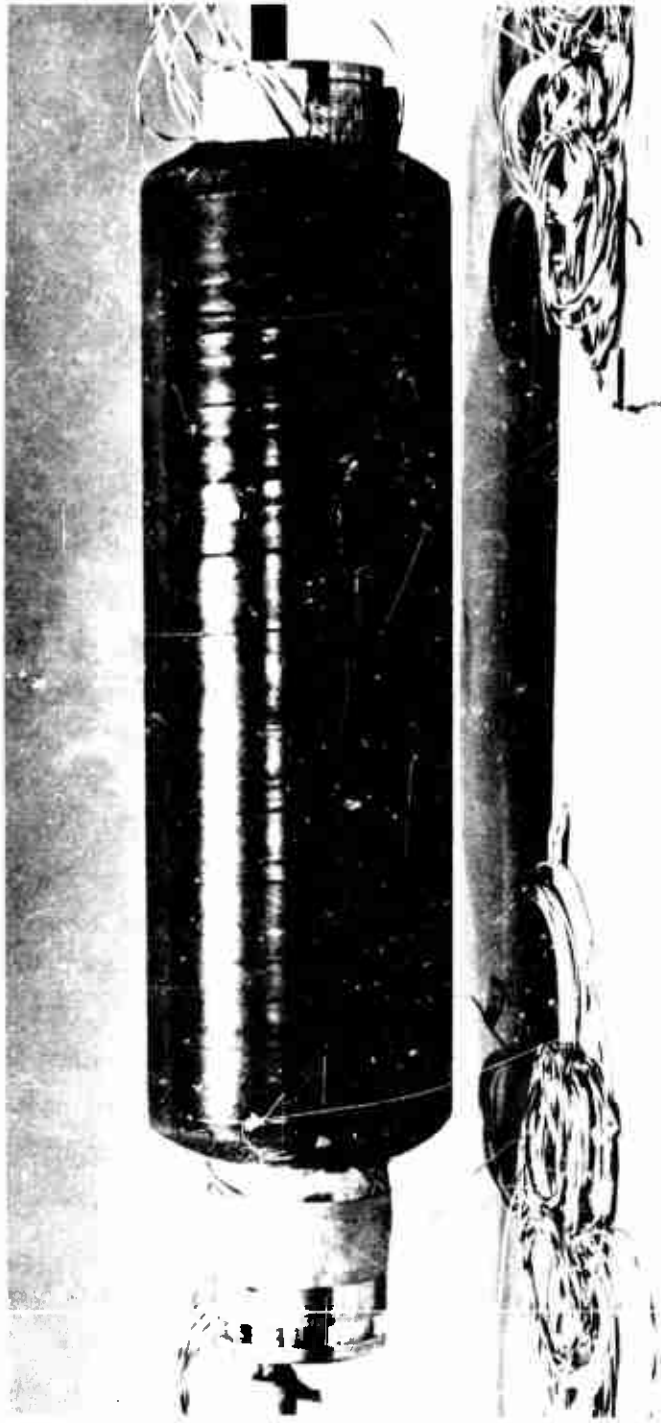


Figure 15. External Pressurization Grain No. 04i-17

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The grain was extensively instrumented with internal hoop gages (refer to Fig. 14 for locations), longitudinal displacement transducers (on ID and OD), and an ID change transducer. Calibration and recording procedures were similar to those previously described.

The grain was maintained in position in the flange by a cradle consisting of two rods and a support. This cradle was constructed so that it would automatically be released when pressure sufficient to support the weight of the grain was applied. The grain was then lowered into the chamber (refer to Fig. 16) and the top flange was bolted in place. The test procedure involved gradually increasing pressure until a maximum was attained.

RESULTS

The instrumentation revealed that an initial linear functional relationship existed between strain and pressure. When the pressure reached 450 psi, the instrumentation indicated that the grain had buckled. After buckling, complete axisymmetry of the strains was lost. Consequently, the readings from the gages and displacement transducers were no longer meaningful.

After this initial instability, the grain continued to withstand increasing applied pressure. The pressure was increased until complete collapses occurred. The maximum pressure reached was 1920 psi. Figure 17 shows the failed grain after the removal of the protective rubber coating. The three light areas are regions where the reinforced propellant shell was being extruded into the core perforations. The light lines running longitudinally are creases caused by the collapse of the grain wall.

The grain was then sectioned for internal examination. A cut was first made at the middle of the grain normal to the grain axis. The two resulting pieces were then cut longitudinally. Figure 18 shows a transverse section of the grain. Extrusion of both the wire and the matrix of the outer shell

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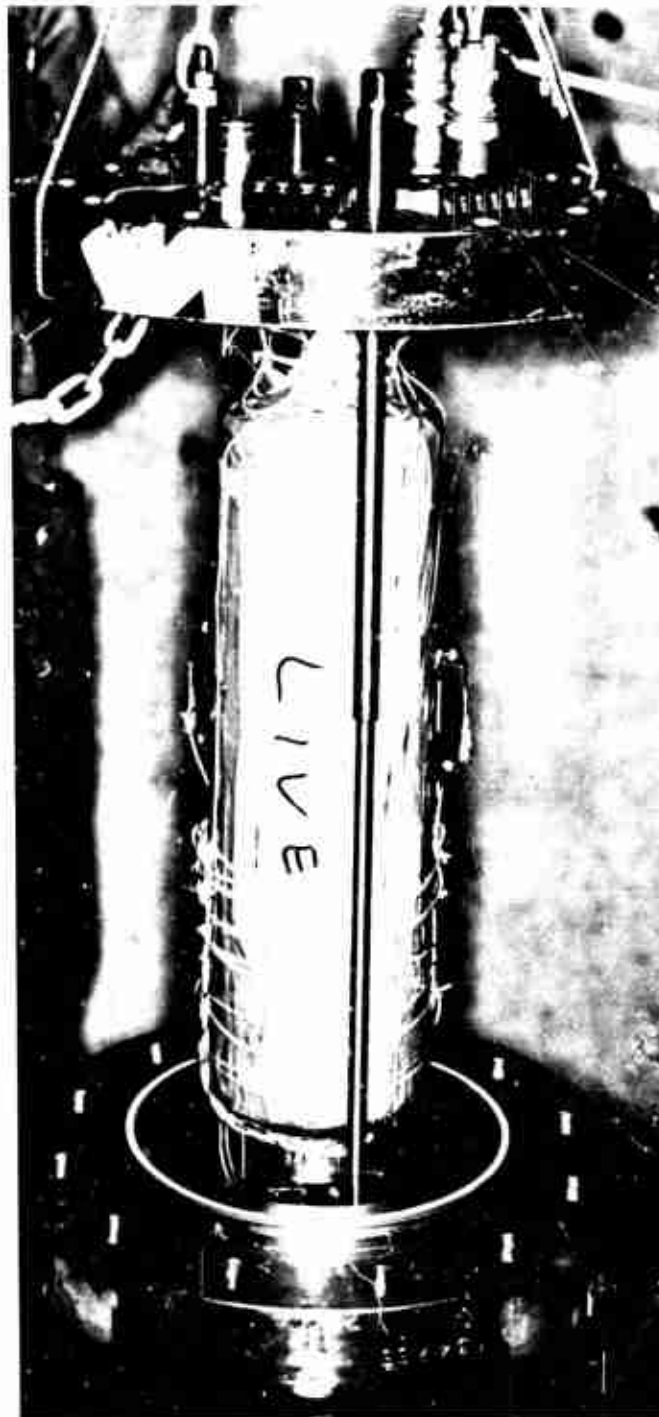


Figure 16. Perforated Core Grain Prepared for External Pressurization Test

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Figure 17. Perforated-Core Grain After Buckling Failure

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into the perforation is clearly visible. Shear failure lines (light lines) are apparent in the matrix above the perforation. A delamination between the shell and core is observable to the left of the perforation.

Figure 19 shows a longitudinal grain section. At the bottom of this figure there is a distinct separation of matrix and wires. This, together with posttest examination of the diameter change transducer, indicated that the core suffered almost complete collapse at failure.

One of the main conclusions that can be drawn from the posttest sectioning and analysis is that the perforations in the core were too large. Greater structural integrity would probably have been realized with a design utilizing a larger number of perforations with smaller diameter.

Further studies are required for a better end closure design. The current design caused most of the axial force to be taken by the core. This was substantiated by the longitudinal displacement transducers placed on the ID and OD. A more equitable distribution of axial force would enhance the buckling resistance of this very complex composite grain.

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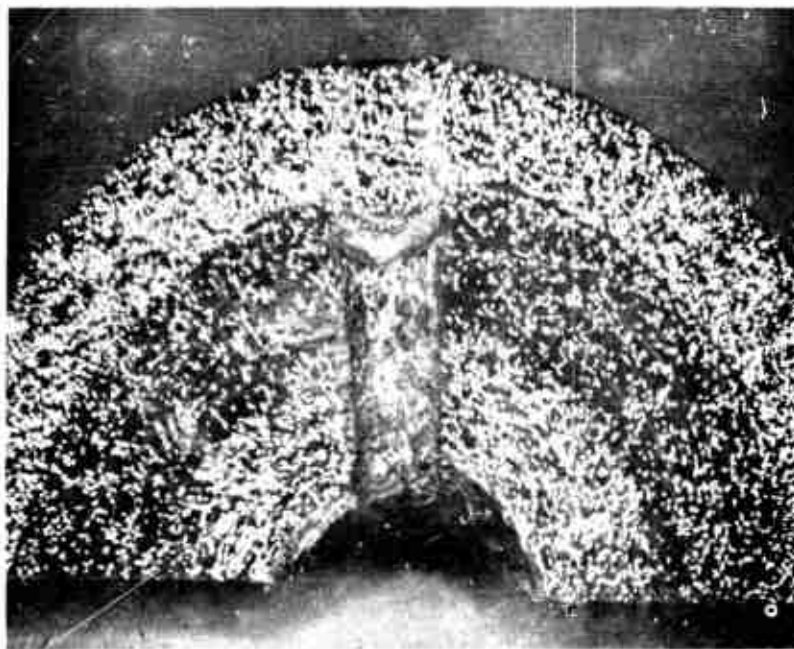


Figure 18. Transverse Section of Failed Grain

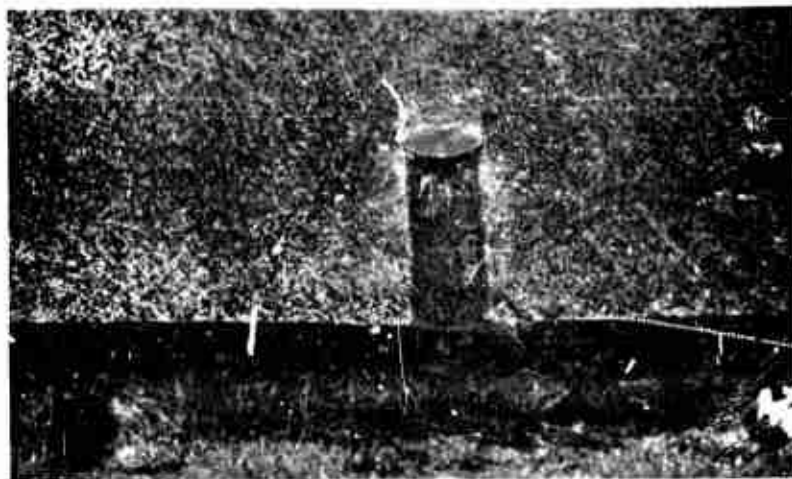


Figure 19. Longitudinal Section of Failed Grain

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SUMMARY AND CONCLUSIONS

Various aspects of the delamination of thick-wall reinforced propellant grains were investigated during this program. An ambient-cure propellant matrix was employed in the fabrication of a thin-wall aluminum wire grain. This demonstrated the compatibility of this matrix system with the helical winding process. Because this matrix cures at room temperature, thick-wall grains would not be subjected to a thermal cycle induced by curing at a high temperature and then bringing to room temperature. In this way, utilization of this matrix in thick-wall RFG may eliminate the delamination problem. This matrix also facilitates prestressing of thick-wall grains in a manner analogous to the autofrettage process.

Another approach to the delamination problem involves providing radial reinforcement. One technique employed with Rocketdyne's usual helical winding process uses radially oriented staples. A grain was fabricated using manually applied ordinary steel office staples. Radiographic examination of the grain revealed that there was large-scale staple motion with very few being radially oriented. This was caused by matrix flow and the action of the compaction roller used during fabrication.

A technique is proposed utilizing short lengths of fine aluminum wire continuously sprinkled over the grain surface during grain fabrication. Then, as the continuous aluminum wires cut into the matrix, the short segments are pulled into the matrix. Portions of the segments will then extend through more than one wire layer and provide a mechanism for radial reinforcement. The use of short segments of aluminum (or 6-micron-diameter graphite) fiber in combination with the ambient-cure matrix may provide the most promising solution to the delamination problem.

A thick-wall grain was made using a spiral wound screen wire technique. This grain was provided with a measure of radial reinforcement by puncturing the screen in such a manner that segments of the screen wire were oriented in a radial direction. This approach was not successful, as was

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evidenced by an obvious delamination which developed while bringing the grain to room temperature. It would appear that the first attempt using this technique simply did not provide sufficient radial reinforcing to prevent delamination.

Experiments were performed to obtain some of the orthotropic characteristics of RFG as a function of winding angle and wire modulus. Aluminum wire grains wound at 54.7 and 30 degrees and a steel wire grain wound at 54.7 degrees were tested in torsion and in uniaxial tension. Thin-wall cylindrical specimens were employed in these tests.

A specially designed thick-wall RFG containing a perforated core was subjected to external pressurization. The grain exhibited a buckling failure after collapse occurred at an external pressure of 1920 psi. A posttest analysis on sections of the failed grain indicated a larger number of smaller diameter holes would provide a superior design. Further end fixture studies were also indicated.

The external pressurization test indicated that wire-reinforced grains can be designed to carry very substantial external pressures as well as the more conventional pressure vessel type of loading. The concept of a perforated core (using smaller perforations than in the grain tested in this program) which is overwound with a shell of reinforced propellant appears to be an attractive idea for a solid propulsion system designed for deep submerged launch applications.

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REFERENCES

1. AFML-TP-65-283, Micromechanics of Fiber-Reinforced Composites, E.L. Alexander, et al., Rocketdyne, a Division of North American Aviation, Inc., Canoga Park, California, November 1965.
2. R-6657, Technical Summary Report, Research on the Response of Reinforced Solid Propellants (U), A. A. Caputo, B. H. Minnich, E. L. Alexander, Nonr 4988(00)FBM, Prog. NR 054-469, Rocketdyne, a Division of North American Aviation, Inc., Canoga Park, California, June 1966, CONFIDENTIAL.
3. R-6497, Technical Summary Report, Reinforced Propellants for Naval Applications (U), E. L. Alexander, et al., Rocketdyne, a Division of North American Aviation, Inc., Canoga Park, California, March 1966, CONFIDENTIAL.

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13. ABSTRACT Research on the structural behavior of wire-reinforced propellants is reported. Values for certain orthotropic material constants which should be useful in grain design have been determined experimentally. The effects of winding angle and wire modulus on the material response were investigated. A problem in delamination of thick-wall wire-reinforced grains was encountered during another Navy program. Various means were therefore investigated in the course of this program to eliminate delamination. Use of an ambient-cure matrix to eliminate thermal stress during the cure cycle and two approaches to adding radial wire reinforcing were explored briefly. The most promising approach appeared to be through use of the new ambient-cure matrix which would be specifically tailored to reinforced propellants. The structural response of an aluminum wire-reinforced grain that was designed to demonstrate deep submergence rocket launch applications was obtained. This grain which embodied a new concept in burning rate control, a perforated inner core, withstood 1920-psi external pressure before complete collapse in the buckling mode. (C)			

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